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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/US97/22877 <b>(22) International Filing Date:</b> 12 December 1997 (12.12.97) <b>(30) Priority Data:</b> 08/778,725 31 December 1996 (31.12.96) US <b>(71) Applicant:</b> KIMBERLY-CLARK WORLDWIDE, INC. [US/US]; 401 North Lake Street, Neenah, WI 54956 (US). <b>(72) Inventors:</b> SOERENS, Dave, Allen; 736 Kensington Road, Neenah, WI 54956 (US). DUSENBERY, Casey, Lynn; Apartment 301, 111 E. Water Street, Appleton, WI 54911 (US). <b>(74) Agents:</b> WARREN, William, L.; Jones & Askew, 37th floor, 191 Peachtree Street, N.E., Atlanta, GA 30303 (US) et al.	<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>	
<b>(54) Title:</b> TEMPERATURE SENSITIVE ADHESIVE COMPOSITION  <b>(57) Abstract</b>  A temperature-triggerable water soluble adhesive composition comprising a poly (vinyl alkyl ether), preferably poly (vinyl methyl ether) and a hydrophobic polymer, preferably a hydrophobic polystyrene resin or an acrylate. The adhesive composition of the present invention retains its integrity at fluid temperatures above approximately 33 °C, but is water soluble at fluid temperatures below approximately 22 °C. The adhesive composition of the present invention may be disposed of by flushing in a conventional toilet.		

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## 5                    **TEMPERATURE SENSITIVE ADHESIVE                          COMPOSITION**

### 10           **TECHNICAL FIELD**

10                    The present invention relates to a flushable adhesive  
composition that may be disposed of by flushing in a conventional  
toilet. More particularly, the present invention relates to a  
temperature-triggerable water soluble adhesive composition  
15                    comprising a poly (vinyl alkyl ether), preferably poly (vinyl methyl  
ether) and a hydrophobic polymer, preferably a hydrophobic  
polystyrene resin or an acrylate. The adhesive composition of the  
present invention retains its integrity at fluid temperatures above  
approximately 33° C., but is water soluble at fluid temperatures  
20                    below approximately 22° C.

### 20           **BACKGROUND OF THE INVENTION**

25                    The term "adhesive" as used herein encompasses the  
term adhesive and its art accepted usages. For example, the term  
adhesive includes, but is not limited to, the material that causes one  
surface to adhere to another like or unlike surface.

30                    The term "flushable" as used herein means capable of  
being flushed in a conventional toilet and being introduced into a  
conventional sewage system such as a municipal sewage system, an  
industrial septic tank system and a residential septic tank system,  
without causing an obstruction or blockage in the conventional toilet  
or conventional sewage system.

35                    The term "flushable adhesive" as used herein means the  
temperature-triggerable water soluble adhesive of the present  
invention.

                      The term "flushable product" as used herein refers to  
products comprising or containing the flushable adhesive of the

present invention and includes, but is not limited to, adhesives, tapes, labels, sheets, papers, tissues, structural members, nonwoven fabric articles, woven fabric articles and combinations thereof. Flushable articles include, but are not limited to, infant care articles such as  
5      diapers, protectors, bibs and wipes; child care articles such as training pants, protectors, bibs and wipes; adult care articles such as diapers and incontinence pads; feminine care articles such as pads and tampons; medical care articles such as bed coverings, wound dressings, ostomy bags, wipes and gowns; surgical care articles such  
10     as table coverings, drapes, wound dressings and gowns; household articles such as wipes; insulation; and packaging materials.

The term "water dispersible" as used herein means an adhesive film which, when exposed to a fluid at a temperature of approximately 22° C. for approximately 2 minutes, dissolves or  
15     fragments into pieces all of which pass through a 20 mesh screen.

The term "water disintegratable" as used herein means an adhesive film which, when exposed to a fluid at a temperature of approximately 22° C. for approximately 2 minutes, disintegrates or  
20     fragments into pieces some of which will pass through a 20 mesh screen.

The term "water weakened" as used herein means an adhesive film which, when exposed to a fluid at a temperature of approximately 22° C. for approximately 5 minutes, loses rigidity and  
25     will bend without the application of an external force when the film is held horizontally by one corner.

The term "water stable" as used herein means an adhesive film which, when exposed to a fluid at a temperature of approximately 22° C. for approximately 5 minutes, is not water  
30     dispersible, water disintegratable or water weakened.

The term "water soluble" as used herein means an adhesive which is water dispersible or water disintegratable in the presence of fluid having a temperature of approximately 22° C., such  
as the temperature of tap water normally present in a conventional toilet bowl.

The term "water insoluble" as used herein means an adhesive which is water stable in the presence of fluid having a temperature of approximately 33° C., such as the temperature of body waste fluids.

5           The term "temperature-triggerable" as used herein means that the water solubility of an adhesive is temperature dependent. More particularly, the term "temperature-triggerable" means that the flushable adhesive of the present invention is water  
10 insoluble at or above a temperature of approximately 33° C. and is water soluble at or below a temperature of approximately 22° C. The term approximate as used herein with respect to temperature includes temperatures within two degrees of the stated temperature.

Disposable products which are easy to use, relatively inexpensive and sanitary are a great convenience. However, as the  
15 closing of solid waste landfills preclude dumping and as clean air standards preclude incineration, disposal of these products has become a problem. An alternative to dumping and incineration is the use of flushable products which can be disposed of by flushing down a conventional toilet into a conventional sewage system. To be  
20 flushable, a product must meet two criteria. First, it must have sufficient wet strength for its intended use. Second, it must be water soluble on contact with toilet bowl water.

One approach to producing a flushable product is to limit the size of the product so that it will pass through plumbing without causing blockages. Such products have high wet strength,  
25 but do not disintegrate on contact with tap water in a toilet bowl. A second approach to producing a flushable product is to produce a product which is not itself water soluble but which disintegrates on contact with acidic or alkaline aqueous solutions or in the presence of specific enzymes. Such products have high wet strength, but require  
30 the addition of acidic, alkaline or enzymatic material to enable their disposal in a conventional sewage system. A third approach to producing a flushable product is to adhere a product together with a salt sensitive adhesive. However, some salt-sensitive adhesives precipitate in the presence of high calcium ion concentrations. As  
35

the calcium content of normal tap water varies tremendously among geographic locales (1982-1983 Analysis of Metropolitan Water Supplies), in locales having high calcium containing water, products produced using calcium adhesives may not, in fact, be flushable. For a useful flushable adhesive, the adhesive used should be flushable in the tap water of any geographic locale so that the flushable product disperses or disintegrates on contact with the tap water.

However, even if a flushable adhesive and a flushable product containing the flushable adhesive are water soluble in any geographic locale, to be useful the flushable product must also have sufficient wet strength for its intended use. Sufficient wet strength for its intended use and dispersion or disintegration on contact with toilet bowl water is particularly difficult to achieve for adhesives in disposable products designed to contact body fluids. This difficulty results from the similarity in composition of body fluids and of tap water. This is illustrated in Table 1. which compares the pH and ionic composition of the body fluid urine and of tap water.

Parameter	Infant Urine	Tap Water
pH	5.8-8.5	5.0 10.5
Calcium	4-182 ppm	0-145 ppm
Magnesium	6-333 ppm	0-120 ppm
Sodium	12-6,200 ppm	1-198 ppm
Chloride	190-10,320 ppm	0-540 ppm
Sulfate	23-3,514 ppm	0-572 ppm

There is, however, a distinct difference between the temperature of body fluids and of tap water. For example, the skin temperature within the area covered by a disposable diaper averages approximately 34.3° C. (Jordan, W.E. and Bailey T.L. 1982. "Factors influencing infant diaper dermatitis" in *Neonatal Skin*, H. Maibach and K. Boisit, Eds. Marcel Dekker, Inc. New York), whereas the temperature of toilet bowl water is approximately 20° C. - 25° C. This temperature difference between body waste fluids and toilet bowl water provides an opportunity to exploit temperature as a trigger wherein the adhesive is water insoluble at one temperature

and water soluble at another temperature. Such a temperature-triggerable water soluble adhesive can be used alone or as a means to adhere one substrate to another like or unlike substrate. Such a flushable adhesive would enable a flushable product to disperse or disintegrate into its component parts each of which can be disposed of in the most environmentally efficient and cost effective manner.

Therefore, what is needed is a non-toxic, temperature-triggerable water soluble adhesive which has sufficient wet strength for its intended use at one temperature and which is water soluble at another temperature. That is a flushable adhesive which is water insoluble at the temperature of body waste fluids, but is water soluble at the temperature of normal tap water and, therefore, is flushable into conventional sewage systems.

#### SUMMARY OF THE INVENTION

The flushable adhesive of the present invention comprises a temperature-triggerable water soluble polymer, wherein the polymer is a poly (vinyl alkyl ether), preferably a poly (vinyl methyl ether) (PVME) obtained as Amobond (Amoco Chemical Co.) and a means for altering the temperature at which the PVME is water soluble, the means comprising a hydrophobic polymer, preferably a polystyrene resin or an acrylate, admixed with the PVME in an amount sufficient to render the admixed PVME and the hydrophobic polymer water insoluble in the presence of fluid having a temperature above approximately 33° C., but water soluble in the presence of fluid having a temperature below approximately 22° C. The flushable adhesive composition of the present invention is used alone and to adhere one substrate to another like or unlike substrate.

Accordingly, it is an object of the present invention to provide a flushable adhesive which is water insoluble in fluid having a temperature above approximately 33° C., but is water soluble in fluid having a temperature below approximately 22° C.

It is another object of the present invention to provide a flushable adhesive that disintegrates in tap water regardless of geographic variations in tap water salt concentration.



It is another object of the present invention to provide a flushable adhesive that is non-toxic and that is non-irritating to the skin.

5 It is another object of the present invention to provide a permanent adhesive that is converted to a removable adhesive by a change in the temperature of the fluid to which the adhesive is exposed.

10 It is another object of the present invention to provide an adhesive which retains satisfactory adhesion properties in the presence of changes in ambient humidity.

It is another object of the present invention to provide a flushable adhesive as a part of a disposable product that can be flushed down a toilet.

15 It is another object of the present invention to provide a flushable adhesive as a part of a disposable product that can be flushed down a toilet without the addition of dissolving chemicals.

It is another object of the present invention to provide a flushable adhesive for use in infant care articles.

20 It is another object of the present invention to provide a flushable adhesive for use in child care articles.

It is another object of the present invention to provide a flushable adhesive for use in adult care articles.

It is another object of the present invention to provide a flushable adhesive for use in feminine care articles.

25 It is another object of the present invention to provide a flushable adhesive for use in medical care articles.

It is another object of the present invention to provide a flushable adhesive for use in surgical care articles.

30 It is another object of the present invention to provide a flushable adhesive for use in household articles.

It is another object of the present invention to provide a flushable adhesive for use in insulation.

35 It is another object of the present invention to provide a flushable adhesive for use in packaging material.

## BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 depicts the water-uptake of films prepared from 100% PVME, from 75% PVME + 25% Piccotex-100 and from 50% PVME + 50% Piccotex after 1, 4, 9 and 16 minutes exposure to water at 20° C.

Fig. 2 depicts the water-uptake of films prepared from 100% PVME, from 75% PVME + 25% Piccotex-100 and from 50% PVME + 50% Piccotex after 1, 4, 9 and 16 minutes exposure to synthetic urine at 35° C.

Fig. 3 depicts the water-uptake of films prepared from 75% PVME + 25% Kristalex 5140 after 1, 4, 9 and 16 minutes exposure to water at 20° C., to water at 35° C. and to synthetic urine at 35°C.

Fig. 4 depicts the water-uptake of films prepared from 100% PVME after 1, 4, 9 and 16 minutes exposure to water at 20° C., to water at 35° C. and to synthetic urine at 35° C.

## DETAILED DESCRIPTION OF THE INVENTION

The temperature-triggerable water soluble adhesive of the present invention comprises two components. The first component is a poly(vinyl alkyl ether), preferably a poly (vinyl methyl ether) (PVME). The second component is a hydrophobic polymer, preferably a hydrophobic polystyrene resin or an acrylate.

The PVME for use in this invention is a polymer which is soluble in water below 33°-35° C. and insoluble in water above 33°-35° C., which is the cloud point temperature (T<sub>LCST</sub>) for PVME. The closeness of the T<sub>LCST</sub> of PVME to the temperature of body waste fluids makes PVME unsuitable for use as an adhesive in flushable products designed to contact the body of a human or animal. In addition, PVME is characterized by a low glass-transition temperature (T<sub>g</sub>) of -21° C. and, therefore, a pronounced tendency to "cold flow". That is, PVME tends to flow away from the bonding site on storage at or below room temperature. This "cold flow" tendency makes PVME unsuitable for use as an adhesive in products which must be stored for a length of time.

To use PVME as a temperature-triggerable water soluble adhesive either alone or in flushable products intended to contact the body of a human or animal, it is necessary to reduce its solubility in fluid at temperatures of 33°-35° C. without significantly decreasing its solubility in fluids at 22° C.

The hydrophobic polymers for use in this invention are more water insensitive than PVME and are characterized by positive Tgs of preferably between approximately 50° C. and 95° C., more preferably between approximately 55° C. and 92° C., and most preferably between approximately 60° C. and 91° C. As expected, when a hydrophobic polymer is blended with PVME, the composition is less sensitive to water above 33° C. than is PVME alone. However, totally unexpectedly, when a hydrophobic polymer is blended with PVME, the composition is more sensitive to water below 22° C. than is PVME alone. That is, blends of PVME and a hydrophobic polymer are more inert to fluids at 33° C. than PVME alone and therefore provide more wet strength than PVME alone. Even more importantly, blends of PVME and a hydrophobic polymer absorb water faster at 22° C. than PVME alone and therefore provide a more flushable adhesive than PVME alone. Moreover, the flushable adhesive of the present invention comprising PVME and a hydrophobic polymer is characterized by an increase in Tg over that of PVME alone and a decrease in the "cold flow" over that of PVME alone. This increase in Tg and decrease in "cold flow" make the PVME-hydrophobic polymer flushable adhesive of the present invention an unexpectedly superior flushable adhesive.

The PVME for use in the present invention can be amorphous or isotactic. The PVME preferably has a number average molecular weight of 40,000 to 300,000 Daltons, more preferably a number average molecular weight of 80,000 to 200,000 Daltons and most preferably a number average molecular weight of 120,000 to 160,000 Daltons. Preferably, the PVME comprises between approximately 10% and 90% by weight (grams/100 grams), more preferably, between approximately 25% and 80% by weight and most preferably between approximately 50% and 75% by weight of

the temperature-triggerable water soluble adhesive composition of the present invention.

The hydrophobic polymers for use in the present invention include, but are not limited to, polystyrene copolymers and acrylates. Polystyrene copolymers include, but are not limited to, a polystyrene oligomer such as Piccolastic D150 (Hercules Inc.), an alpha-methyl styrene such as Kristalex 3100 (Hercules Inc.) or Amoco Resin 18-290 (Amoco Chemical Co.), an aromatic copolymer such as Kristalex 5140 (Hercules Inc.), a terpene phenol such a Piccofyn A135 (Hercules Inc.), a poly (vinyl toluene-co-alpha methyl styrene) copolymer such as Piccotex 100 (Hercules Inc.), and a pentaerythritol ester of polymerized resin such as Pentalyn C. (Hercules Inc.). Preferably, the hydrophobic polystyrene resin comprises between approximately 10% and 90% by weight (grams/100 grams), more preferably between approximately 20% and 75% by weight and most preferably between approximately 25% and 50% by weight of the flushable adhesive of the present invention. Acrylates include, but are not limited to, a poly (ethyl acrylate-co-methacrylic acid) copolymer such as ASE-75 (Rohm & Haas) and a cross-linked poly (ethyl acrylate-co-methacrylic acid) copolymer such as ASE-60 (Rohm & Haas). Preferably, the acrylate comprises between approximately 20% and 65% by weight (grams/100 grams), more preferably between approximately 25% and 55% by weight and most preferably between approximately 40% and 50% by weight of the flushable adhesive of the present invention.

The flushable adhesive of the present invention can contain optional non-hydrophilic additives including, but not limited to, anti-oxidants, pigments, and colorants.

The flushable adhesive of the present invention can be used alone or can be applied to a substrate including, but not limited to, tape, film, paper, structural elements, woven fabric, nonwoven fabric and combinations thereof. Methods of applying the flushable adhesive of the present invention to a substrate include, but are not limited to, rolling, dipping, dotting, printing, extruding, spraying and other methods known to those skilled in the art.

The flushable adhesive of the present invention is prepared by blending PVME and a hydrophobic polymer until a homogeneous mixture is obtained using any of the various methods known in the art. Such methods include, but are not limited to, mixing a solution of PVME and a solution of a hydrophobic polymer in a common solvent and evaporating the solvent or hot melt mixing the PVME and the hydrophobic polymer. The hot melt method used may be any of the methods known to those skilled in the art. It is to be understood that the precise temperature to be used in the hot melt method will depend on the melting point of the PVME and of the hydrophobic polymer.

The following examples illustrate a variety of the flushable adhesives that can be made within the scope of the present invention.

15

#### EXAMPLE 1- FLUID UPTAKE

Fluid uptake is used to evaluate the performance of the flushable adhesive of the present invention. Fluid uptake is important because the flushable adhesive of the present invention must be water insoluble in the presence of fluid having a temperature above approximately 33° C., but water soluble in the presence of fluid having a temperature below approximately 22° C.

To measure fluid uptake one-half inch squares of adhesive film are weighed and are immersed in water at 22° C. and 35° C. and in synthetic urine at 35° C. for 1, 4, 9 and 16 minutes. Weight gain is measured and fluid uptake is defined as [final weight - initial weight]/initial weight.

PVME - Adhesive film is prepared by dissolving 100% by weight of PVME having a number average molecular weight of 122,000 Daltons in ethyl acetate. The dissolved PVME is poured into a silicone rubber mold, the solvent is allowed to evaporate for 3 days at room temperature and the film pressed at 150° F. for 5 minutes. The resulting film is a 0.43 cm, bubble-free, uniform, smooth film.

PVME+PICCOTEX 100 BLENDS - Adhesive films are prepared by dissolving 75% by weight of PVME having a number average molecular weight of 122,000 Daltons in ethyl acetate and 25% by weight of Piccotex 100 in ethyl acetate and by dissolving 50% by weight of PVME having a number average molecular weight of 122,000 Daltons in ethyl acetate and 50% by weight of Piccotex 100 in ethyl acetate. The dissolved PVME and the dissolved Piccotex 100 are blended to homogeneity. The PVME-Piccotex 100 blends are poured into a silicone rubber mold, the solvent is allowed to evaporate for 3 days at room temperature and the films are pressed at 150° F. for 5 minutes. The resulting films are 0.43 mm in thickness, bubble-free, uniform and smooth.

Figure 1. shows the water uptake of a 100% PVME film, a 75% PVME+25% Piccotex 100 film and a 50% PVME+50% Piccotex 100 film after 1, 4, 9 and 16 minutes of immersion in water at 22° C.

Figure 2. shows the fluid uptake of a 100% PVME film, a 75% PVME+25% Piccotex 100 film and a 50% PVME+50% Piccotex-100 film after 1, 4, 9 and 16 minutes of immersion in synthetic urine at 35° C.

These data show that adhesive films prepared from blends of 25% and 50% of the hydrophobic polystyrene resin Piccotex 100 with 50% and 75% PVME show an increase in fluid uptake at 22° C. and reduction in fluid uptake at 35° C. over that of 100% PVME alone.

PVME+KRISTALEX 5140 BLENDS - An adhesive film is prepared by dissolving 75% by weight of PVME in ethyl acetate and 25% by weight of Kristalex 5140 in ethyl acetate. The dissolved PVME and the dissolved Kristalex 5140 are blended to homogeneity. The PVME+Kristalex 5140 blend is poured into a silicone rubber mold, the solvent is allowed to evaporate for 3 days at room temperature and the films are pressed at 150° F for 5 minutes. The resulting films are 0.43 mm in thickness, bubble-free, uniform and smooth.

Figure 3 shows the fluid uptake of a 100% PVME adhesive film after 1, 4, 9 and 16 minutes of immersion in water at 20° C. and after 1, 4, 9 and 16 minutes of immersion in water or in synthetic urine at 35° C.

5                Figure 4 shows the fluid uptake of the 75% PVME+25% Kristalex 5140 adhesive film after 1, 4, 9 and 16 minutes of immersion in water at 22° C. and after 1, 4, 9 and 16 minutes of immersion in water or in synthetic urine at 35° C.

10              These data show that blending 25% of the hydrophobic polystyrene resin Kristalex 5140 with 75% PVME increases fluid uptake at 20° C.-22° C. and reduces fluid uptake at 35° C. over that of 100% PVME alone.

## EXAMPLE 2 - FLUID UPTAKE

15              Adhesive films are prepared by mixing from 50% to 100% by weight of PVME and from 25% to 50% by weight of the hydrophobic polystyrene resins Piccotex 100, Piccolastic D 150 and Amoco Resin 18-290 and heating the mixture in a Haake Rheomix 600 to a temperature at which it is a flowable fluid (200° C. to 20              350° C.). Each PVME-hydrophobic polystyrene resin hot blend is mixed for approximately 10 minutes until homogeneous, filtered if needed, cooled and pressed with a 0.13 mm shim in a Dake press to form a film. Each film is tested for water sensitivity at 22° C. and at 35° C. and for urine sensitivity at 35° C.

25              Table 2 compares the percent fluid uptake of the 100% PVME adhesive film with the fluid uptake of the PVME-hydrophobic polystyrene resin adhesive films after 9 minutes of immersion in water at 22° C. and at 35° C. and in synthetic urine at 35° C.

**Table 2**  
**% UPTAKE (9 minute immersion)**

BLEND	PVME	25%P-100 75% PVME	50%P-100 50% PVME	25% Amoco 75% PVME	50% Amoco 50% PVME	25% PS 75% PVME	60% PS 60% PVME
water @ 22° C.	.38%	.92%	.43%	.83%	.52%	.55%	.39%
water @ 35° C.	.35%	.24%	.08%	.24%	.03%	.23%	.06%
synthetic urine @ 35° C.	.29%	.27%	.02%	.13%	.04%	.17%	.12%

P-100 = Piccotex 100

PS = Piccolastic D150

Amoco = Amoco Resin 18-290

These data show that adhesive films prepared by blending 25% to 50% of the hydrophobic polystyrene resins Piccotex 100, Piccolastic D150 and Amoco Resin 18-290 with 50% to 75% PVME show reduced fluid uptake at 35° C. and increased fluid uptake at 22° C. over that of adhesive films prepared from 100% PVME.

Examples 1 and 2 demonstrate that flushable adhesives of the present invention comprising PVME and a hydrophobic polystyrene resin take up less fluid at 35° C. than PVME alone and absorb more fluid at 22° C. than PVME alone. These unexpected results show that the adhesive composition of the present invention has both greater wet strength at 35° C. than PVME alone and greater water solubility at 22° C. than PVME alone.

### EXAMPLE 3 - GLASS TRANSITION TEMPERATURE

Glass transition temperature (T<sub>g</sub>) is the temperature at which a polymer changes from its glass-like state to a rubber-like state due to increased molecular motion. The T<sub>g</sub> of a polymer blend is an indicator of the miscibility of the polymers in the blend. T<sub>g</sub> is determined by the step change in heat capacity versus temperature measured using a TA instruments 2910 Differential Scanning



Calorimeter. The Tg is chosen as the midpoint of the change in heat capacity at a heating rate of 20 degrees per minute.

Table 3A shows the Tg of PVME, the Tgs of the hydrophobic polystyrene resins Piccolastic D150, Kristalex 5140, Piccotex 100 and the Tg of the acrylate ASE-60. Figure 3B shows the Tgs of blends of 50% and 75% PVME with 25% and 50% of Piccolastic D150, Kristalex 5140, Piccotex 100, Pentacyn C and ASE-60.

Table 3A

Tg of Starting Material	Tg (°C.)
PVME	-21
Piccolastic D150	69
Kristalex 5140	91
Piccotex 100	60
ASE-60	76

Table 3B

Tg of Blends	Tg (°C.)*
75% PVME + 25% Piccolastic D-150	-19
50% PVME + 50% Piccolastic D-150	-14
25% PVME + 75% Kristalex 5140	-18
75% PVME + 25% Piccotex-100	-16
50% PVME + 50% Piccotex-100	-7
75% PVME + 25% Pentacyn C	-7
75% PVME + 25% ASE-60	-15
50% PVME + 50% ASE-60	3

\*Tgs are  $\pm 10^\circ\text{C}$ .

Table 3A and in Table 3B show that blending PVME with the representative hydrophobic polystyrene resins Piccolastic D150, Kristalex 5140, Piccotex 100 and the Tg of with the representative acrylate ASE-60 raises the Tg of the PVME and lowers the Tgs of the representative hydrophobic polystyrene resins and of the representative acrylic to a single Tg value in-between that of the representative hydrophobic polystyrene resins and of the representative acrylic used. Moreover, the increase in the Tgs of the PVME+hydrophobic polystyrene resin blends and of the PVME+acrylate blends over that of 100% PVME indicates that the blends have less of a tendency to "cold flow" than does the PVME alone.

#### EXAMPLE 4 - DIFFUSIVITY

Diffusivity testing is used to determine the effect of water vapor on the flushable adhesive of the present invention. This is important because the flushable adhesive and products containing the flushable adhesive must be stored, shipped and worn in various humidities.

To measure diffusivity, half inch squares of a film of the flushable adhesive of the present invention are put in a desiccator at high humidity for 7 days. The humidity is created by placing a slurry of  $K_2SO_4$  in the desiccator which gives a relative humidity of 97% at room temperature. The weight gain is measured periodically and  $R(t)$  is calculated, plotted versus the square root of time and the slope  $K$  is found through a regression. The diffusivity coefficient  $D$  is calculated using the equation  $D = \pi/16 \cdot K^2 \cdot \delta^2$ , where  $\delta$  is film thickness.

Adhesive films are prepared from 100% PVME and from blends of 50% and 75% PVME with 25% and 50% of a representative hydrophobic polystyrene resin according to the methods of Examples 1 and Example 2. PVME+acrylate adhesives films are prepared by dissolving 50% by weight of PVME in tetrahydrofluran (THF) and by dissolving 50% by weight of the representative acrylates ASE-60 and ASE-75 in THF. The dissolved

PVME and the dissolved ASE-60 or ASE-75 are blended to homogeneity. The PVME-ASE-60 and PVME ASE-75 blends are poured into silicone rubber molds, the solvent is allowed to evaporate for 3 days at room temperature and the films are pressed at 150° C. for 5 minutes and at room temperature for an additional 12 hours.

Table 3 compares the diffusivity of 100% PVME films to films prepared from blends of from 50% to 75% PVME and from 25% to 50% of representative hydrophobic polystyrene resins and acrylates.

**TABLE 4**  
**Diffusivity Data**

Blend	Thickness (mm)	D
25% Piccolastic	0.45	2.50E-06
50% Piccolastic	0.54	4.66E-06
25% Kristalex 5140	0.41	2.18E-06
25% Piccotex 100	0.43	2.76E-06
50% Piccotex 100	0.34	1.91E-06
25% Pentalyn C	0.45	4.41E-06
25% Amoco 18-290	0.42	6.66E-06
50% Amoco 18-290	0.37	3.13E-06
50% ASE-75	0.41	7.84E-06
50% ASE-60	0.26	2.97E-06
100% PVME*	0.81	3.72E-05

\*PVME is probably not accurate because it became a gel after 3 days.

These data show that adhesive films prepared from blends of 25% and 50% PVME and 25% and 50% of a representative hydrophobic polystyrene resin and of a representative acrylate show reduced diffusivity over adhesive films prepared from 100% PVME after 7 days at 97% humidity.

The flushable adhesives of the present invention provide sufficient wet strength for their intended at the temperature of body fluids, yet disintegrate and disperse at the temperature of normal tap water. The flushable adhesives of the present invention have Tgs at which "cold flow" will not result in the adhesive tending to flow away from the bonding site on storage. The flushable adhesives of the present invention have diffusivity coefficients which enable them to maintain their integrity at high humidities and which allow them to be stored, shipped and worn in different humidities.

The flushable adhesives of the present invention may be used alone, in products composed entirely of flushable components and in products composed of flushable and nonflushable components. In the former case the entire product disintegrates or disperses in normal tap water, whereas in the later case the flushable components disintegrate disperse in normal tap water and the flushable article falls apart into pieces small enough to be flushed into a conventional sewage system without causing obstruction.

It is to be understood that in light of the disclosure of the present invention, modifications and improvements thereon will become readily apparent to those skilled in the art and all such obvious variations are contemplated within the scope of the appended claims.

## CLAIMS

We claim:

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1. An adhesive composition comprising,  
a temperature-sensitive water soluble polymer, wherein  
the polymer is poly (vinyl methyl ether) (PVME) and a means for  
altering the temperature at which the PVME is water soluble, the  
10 means comprising a hydrophobic polymer admixed with the PVME  
in an amount sufficient to render the admixed PVME and the  
hydrophobic polymer water insoluble in the presence of fluid having  
a temperature above approximately 33° C. and water soluble in the  
presence of fluid having a temperature below approximately 22° C.

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2. The adhesive composition of claim 1, wherein the  
hydrophobic polymer is a hydrophobic polystyrene resin.

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3. The adhesive composition of claim 2, wherein the  
hydrophobic polystyrene resin is selected from the group consisting  
of a polystyrene oligomer, an alpha-methyl styrene oligomer, an  
aromatic hydrocarbon resin, a terpene phenol resin, a poly (vinyl  
toluene-co-alpha methyl styrene) copolymer and a pentaerythritol  
ester of polymerized resin.

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4. The adhesive composition of claim 3, wherein the  
hydrophobic polystyrene resin is an aromatic hydrocarbon resin.

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5. The adhesive composition of claim 3, wherein the  
hydrophobic polystyrene resin is a poly (vinyl toluene-co-alpha  
methyl styrene) copolymer.

35

6. The adhesive composition of claim 3, wherein the  
hydrophobic polystyrene resin is a pentaerythritol ester of  
polymerized resin.

7. The adhesive composition of claim 1, wherein the hydrophobic polymer is an acrylate.

5           8. The adhesive composition of claim 7, wherein the acrylate is selected from the group consisting of a poly (ethyl acrylate-co-methacrylic acid) copolymer and a cross-linked poly (ethyl acrylate-co-methacrylic acid) copolymer.

10           9. The adhesive composition of claim 8, wherein the acrylate is a cross-linked poly (ethyl acrylate-co-methacrylic acid) copolymer.

15           10. The adhesive composition of claim 1, wherein the adhesive composition comprises 50% or more by weight of the PVME.

20           11. The adhesive composition of claim 1, wherein the adhesive composition comprises 50% or less by weight of the hydrophobic polymer.

25           12. An adhesive composition comprising, 50% or more by weight of PVME and 50% or less of a hydrophobic polymer selected from the group consisting of a hydrophobic polystyrene resin and an acrylate admixed with the PVME, the admixed PVME and hydrophobic polymer being water insoluble in the presence of fluid having a temperature above approximately 33° C. and being water soluble in the presence of fluid having a temperature below approximately 22° C.

30

uptake vs time  
water at 20 deg C

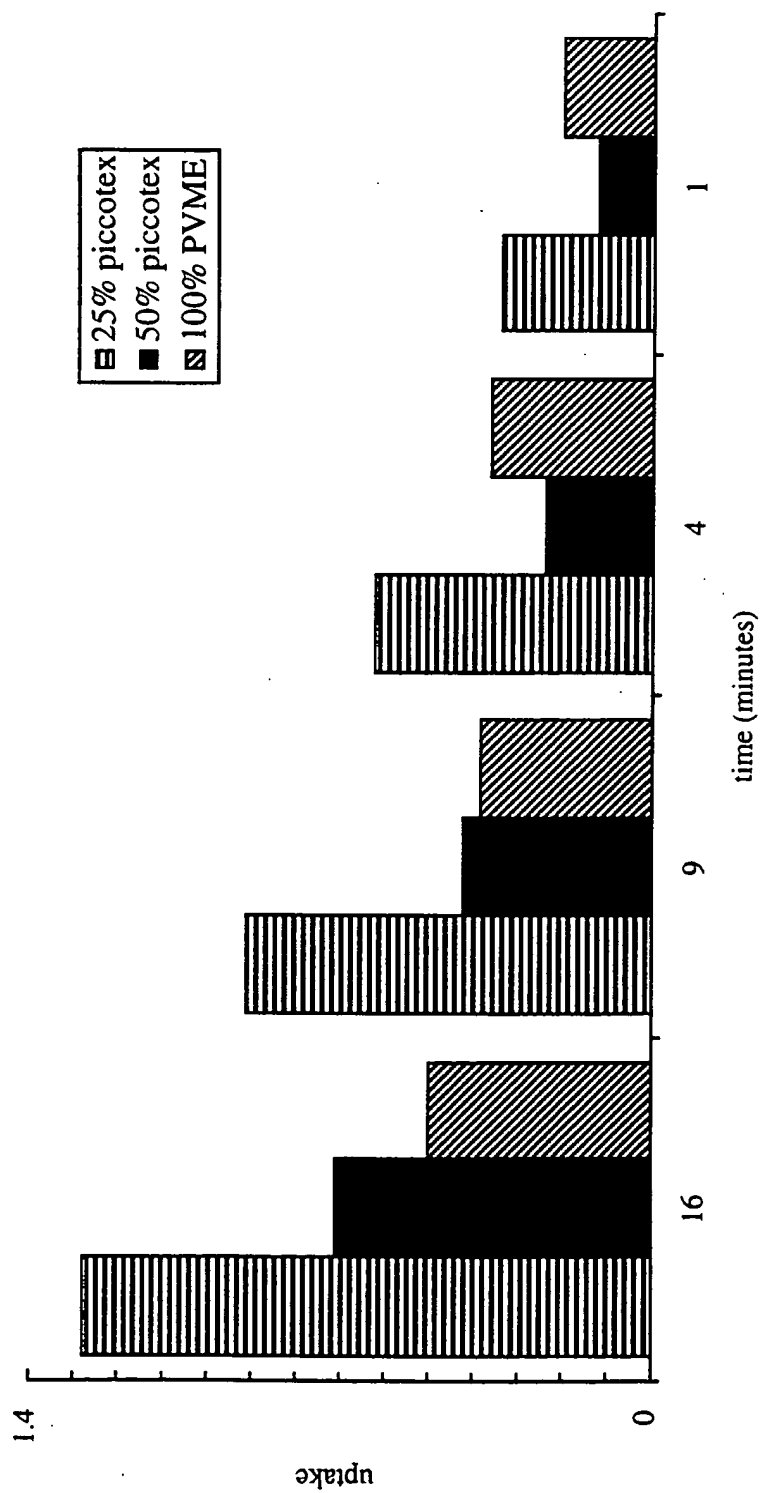


FIGURE 1

uptake vs time  
synthetic urine at 35 deg C

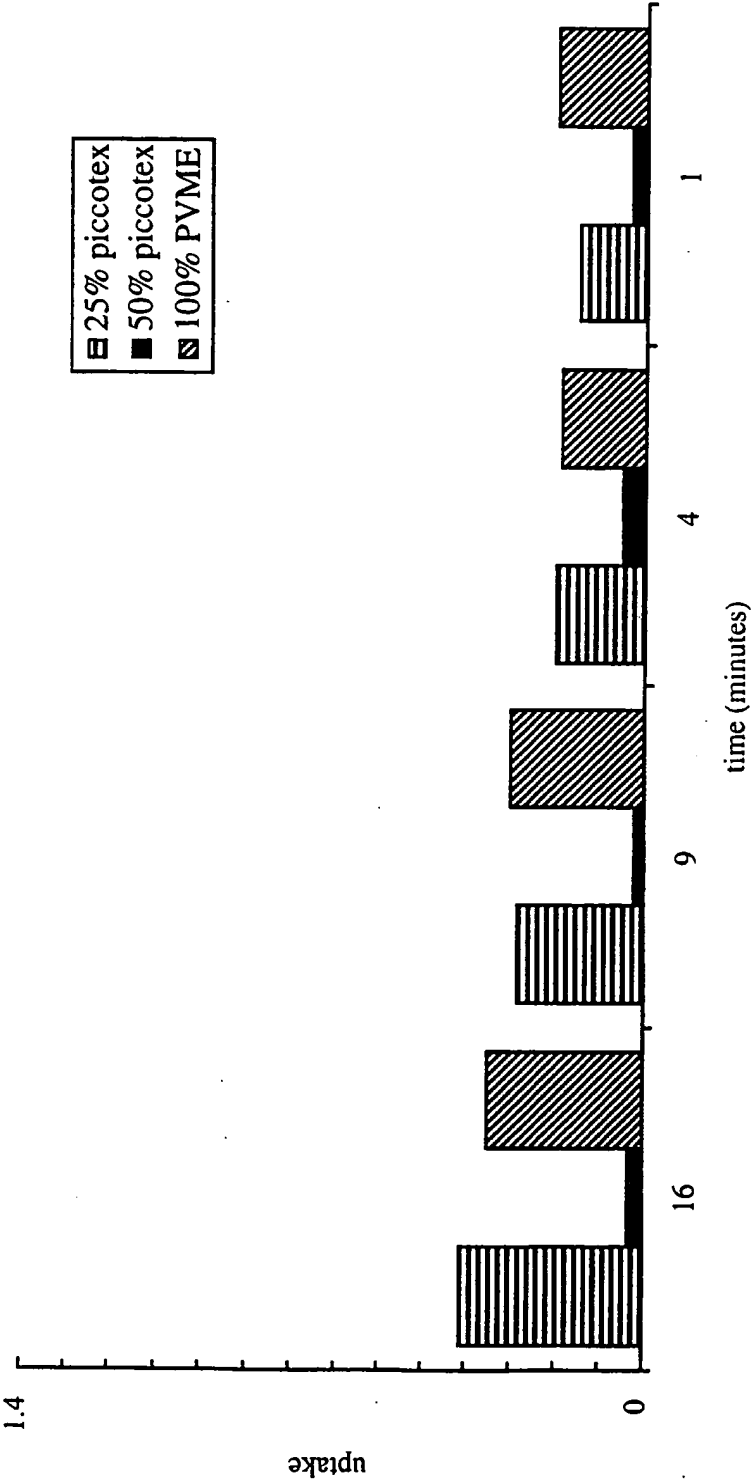


FIGURE 2



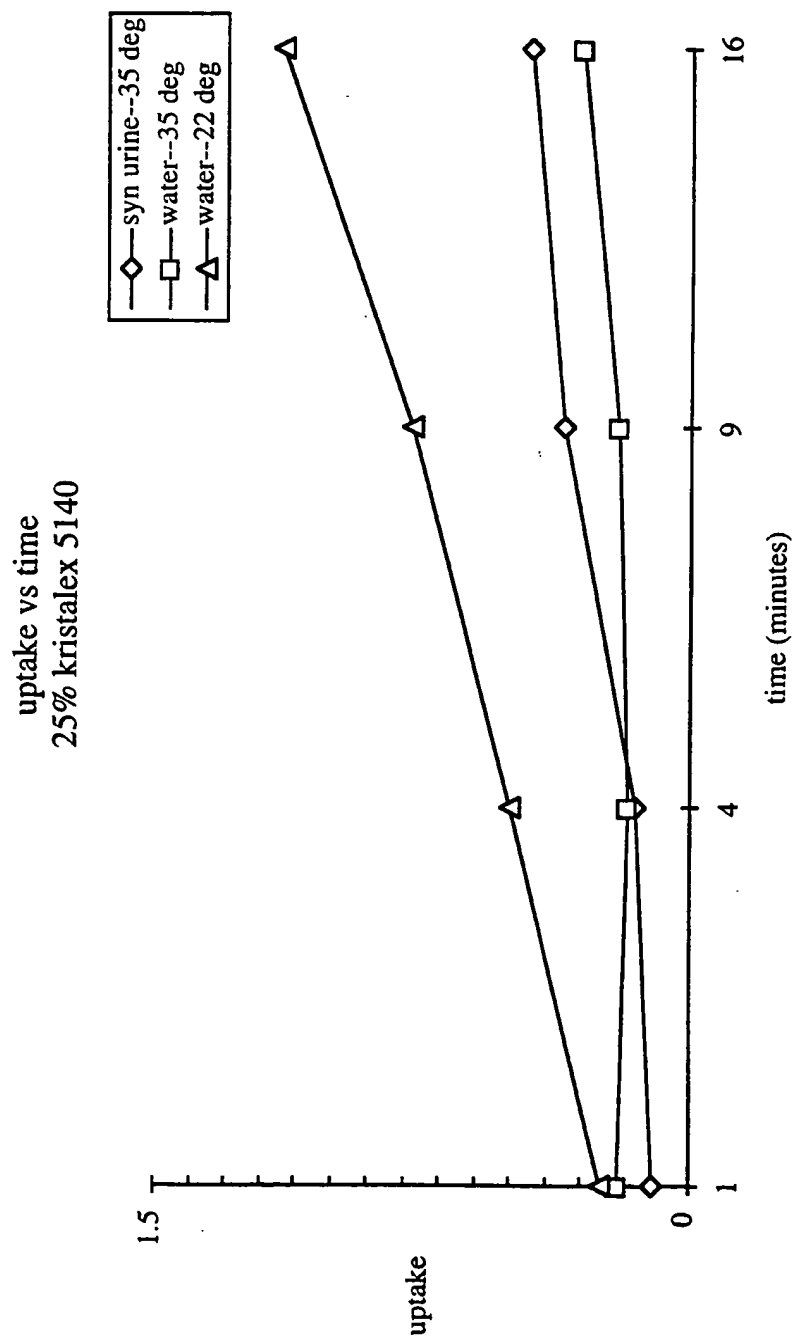


FIGURE 3

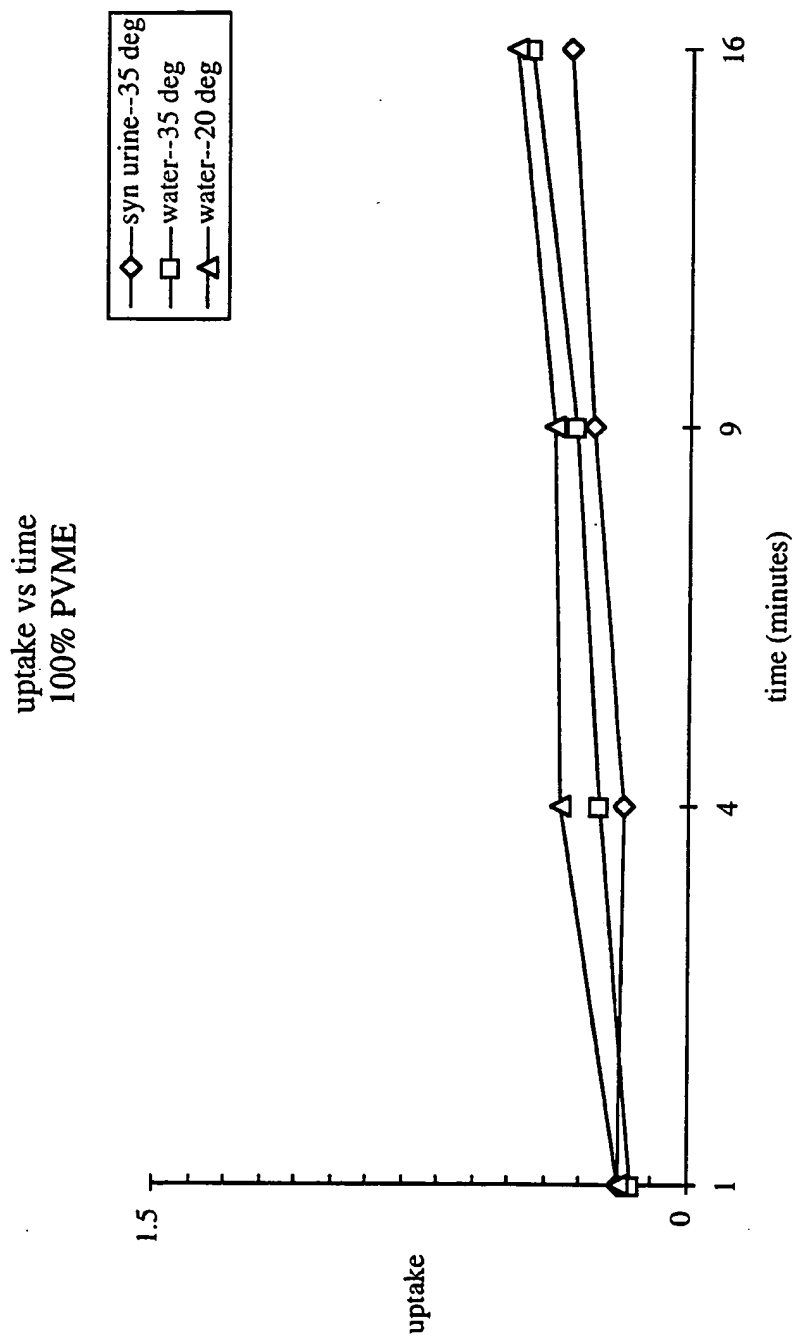


FIGURE 4

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 97/22877

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C09J129/10 A61L15/58 //(C09J129/10,125:02),(C09J129/10,133:00)

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C09J A61L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 644 252 A (SHENFELD R S ET AL) 22 February 1972 see claims; examples 8,10,14 ---	1,2,4, 10-12
X	GB 1 245 410 A (KURAMOTO SANGYO CO.) 8 September 1971 see page 4, line 51 - line 62; claims; examples 1,2,5 ---	1,7, 10-12
X	WO 95 02647 A (FINDLEY ADHESIVES INC) 26 January 1995 see claims; examples ---	1,7
X	WO 95 03361 A (FINDLEY ADHESIVES INC) 2 February 1995 see claims; examples ---	1-3
	-/--	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

2 April 1998

Date of mailing of the international search report

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# INTERNATIONAL SEARCH REPORT

Int. l. Application No

PCT/US 97/22877

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>WO 95 16474 A (KIMBERLY CLARK CO) 22 June 1995  see page 9, line 10 - line 13  see page 13, line 26 - line 30; claims;  example 9</p> <p style="text-align: center;">-----</p>	1

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 97/22877

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 3644252 A	22-02-72	AT 310903 B	15-09-73
		AU 462549 A,B	26-06-75
		AU 3413471 A	05-04-73
		BE 773568 A	31-01-72
		CH 556380 A	29-11-74
		DE 2141912 A	22-02-73
		FR 2157167 A	01-06-73
		GB 1368116 A	25-09-74
		NL 7114129 A	17-04-73
GB 1245410 A	08-09-71	US 3657396 A	18-04-72
WO 9502647 A	26-01-95	US 5527845 A	18-06-96
		AU 6395294 A	13-02-95
		CA 2175359 A	26-01-95
		EP 0711330 A	15-05-96
WO 9503361 A	02-02-95	AU 6405794 A	20-02-95
		CA 2167577 A	02-02-95
		EP 0710264 A	08-05-96
		JP 9503236 T	31-03-97
WO 9516474 A	22-06-95	AU 1336395 A	03-07-95
		BR 9408331 A	19-08-97
		CA 2128483 A	17-06-95
		CN 1142777 A	12-02-97
		DE 4499925 T	19-12-96
		GB 2284820 A	21-06-95
		US 5509913 A	23-04-96
		ZA 9409979 A	22-08-95